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**Effect of 1.33 Mev Gamma Radiation and 0.5 Mev Electrons on the
Mechanical Properties of Graphite Fiber Composites***

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ABSTRACT

Epoxy/graphite fiber, polyimide/graphite fiber, and polysulfone/graphite fiber composites were exposed to 1.33 Mev gamma irradiation and 0.5 Mev electron bombardment for varying periods of time. The effects of the irradiation treatments on the breaking stress and Young's modulus were studied by a three point bending test. Effects were small; indeed, both electron radiation up to 5000 Mrad and gamma radiation up to 350 Mrad resulted in slight increases in both stress and modulus.

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(NASA-CR-169117) EFFECT OF 1.33 Mev GAMMA
RADIATION AND 0.5 Mev ELECTECNS ON THE
MECHANICAL PROPERTIES OF GRAFHITE FIBER
COMPOSITES (North Carolina State Univ.)

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Introduction:

Graphite fiber reinforced composites are light weight, high strength materials that are particularly suitable for space vehicles. Since some space experiments are scheduled over a period of several years, materials used in space may be subjected to substantial quantities of high energy radiation. For this reason, it is important that materials considered for use in these experiments be evaluated with respect to their response to high energy radiation.

Several sets of composite samples, fabricated at NASA Langley Research Center and supplied to us, have been irradiated using 0.5 Mev electrons and 1.33 Mev gamma radiation. The effects of the irradiation treatments on breaking stress and Young's modulus were determined by a three point bending test.

Most of the cosmic radiation in regions near the earth is due to protons [1]. However, significant numbers of both protons and electrons are trapped in the radiation belts around the earth [2,3] and the predominant energy loss in matter of high energy electrons found in geosynchronous orbit is by ionization [3,4]. Similarly, 1.33 Mev gamma radiation would lose most of its energy by ionization through Compton scattering and the photoelectric effect [5]. Therefore, the experimental conditions of radiation exposure of materials to be investigated in this study should provide an excellent simulation of the actual effects of radiation on these materials when used in space applications. Moreover, it has been estimated [6] that the radiation dose for geosynchronous orbit in a thirty year lifetime should be on the order of a thousand Mrad for the 0.056-0.081 cm thickness of the samples we used in the experimental work reported here. This, coupled with the observation that

radiation effects on solid polymers are dose rate independent to first order [7], should indicate that the results described in this paper for doses up to 5000 Mrad should be applicable to the problem of estimating the radiation effects on space vehicles in geosynchronous orbit.

Experimental Procedures:

Studies were made on samples of cured graphite fiber/epoxy composite (T300/5208 Thornel graphite fiber/Narmco 5208 epoxy and AS/3501 graphite fiber/epoxy by Hercules), graphite fiber/polyimide composite (C6000/PNR-15 Celion graphite fiber/polyimide matrix), and graphite fiber/polysulfone composite (C6000/P1700 Celion graphite fiber/polysulfone matrix). The samples were cured and cut at NASA Langley Research Center. Samples of each type were subjected for different periods of time to electron irradiation, and samples of graphite fiber/epoxy were gamma irradiated for different periods of time.

The mechanical tests were made on an Instron using a "three point bending tester" attachment [8]. The specimens, 1.27 by 2.54 cm and 0.056-0.071 cm thick, were tested at a constant rate of elongation, perpendicular to the plane of the composites, at a speed of 0.254 cm/min (0.1 inch/min) and with a span length of 1.40 cm (0.55 inch). The specimens were 4 ply, uniaxially oriented with the preferred axis aligned along the span direction during testing. The ultimate stress and average modulus at each exposure condition were determined by using the standard equations for small bending deformations of elastic bodies [8].

The samples were treated in a vacuum desiccator at 80°C for 7 days, then sealed in aluminum foil (Reynolds Wrap, heavy duty, thickness of 0.025 mm) by first securing the ends of the samples in place with a thin layer of scotch

tape and sealing the edges of the folded aluminum wrap with an epoxy glue (Devcon 5 minute Epoxy®). An open glass tube was inserted prior to sealing the foil to permit a vacuum line to be connected for further vacuum treatment. These packages were placed in a vacuum desiccator at 80°C for at least an additional 4 days; then the glass tube was attached to a vacuum line and heat sealed. The packages then were taken immediately to the electron accelerator and exposed to 0.5 Mev electrons at a current of 8.3 milliamperes. Each package was placed in a ziploc baggie by Dow Chemical (10" by 10") that was prefilled with N₂ to reduce oxidative degradation in case of pin hole leaks, the packages clamped to the conveyor on the accelerator, and passed through the electron beam. Each revolution of the conveyor through the beam resulted in a 10 Mrad dosage. Following the electron irradiation treatment in a 500KV Electron accelerator made by High Voltage Engineering Corporation, the specimens were removed from the packages and placed in open petri containers in a controlled laboratory (r.h. 65%, temperature 20°C) where they remained from 3 to 10 weeks prior to mechanical testing. Following each 400-500 Mrad exposure, the ziploc baggies were replaced and refilled with N₂. After 2500 Mrad, the Al foil in regions of high stress concentrations (sharp bends) showed evidence of degradation and the Devcon epoxy seal on the aluminum foil showed evidence of appreciable discoloration, so the specimen were vacuumed and repackaged in new Al foil as described above.

Two sets of samples, T300/5208 and AS/3501, were exposed to 1.33 Mev gamma radiation for periods up to 500 hours and 1070 hours respectively in a Gamma Cell 220 made by Atomic Energy of Canada. These samples were vacuum desiccated at 80°C for a minimum of three days then placed in the vacuum chamber of the gamma cell (a 1/3 Mrad. per hour Co⁶⁰ source), prevacuumed

treated for 24 hours and exposed to gamma radiation under a continuous vacuum (approximately 5×10^{-6} m Hg.). The samples, after gamma exposure, were tested using the same procedures for equilibration and mechanical testing as described above.

Results and Discussion

The theoretical prediction of stopping power of high energy electrons is given as by Segre [5].

However, due to a secondary scattering, the effective irradiation dosage in a material will first increase and then is followed by a rapid monotonic decrease in radiation dosage [9]. Since the electron accelerator used in our experiments is designed to pass samples across the beam twice during each revolution of the conveyor (once on the front side of the sample and once on the back side), the approximate dosage experienced by the composite specimens as a function of penetration depth is as shown in Fig. 1. The density of the composite is about 1.55 g/cm^3 . The effective thickness of the baggie and aluminum foil is approximately 0.013 cm. Thus, the effective radiation dosage in the center of the specimen is approximately 40% higher than on the edge.

The load deformation curves of the three point bending tests were approximately linear in all cases and the deflections small so that equations for small bending deformations give excellent approximations of ultimate stress and Young's modulus [8]. Effects of electrons and gamma radiation on these parameters for the samples investigated are summarized in Figs. 2-5. A minimum of 10 replication measurements were made for each exposure condition. All samples exposed to 5000 Mrad of 0.5 Mev electrons showed a slight increase in both stress and modulus compared with the control. In each case,

the increases were essentially monotonic. The increases at 5000 Mrad to the control were 13%, 10%, and 11.5% in stress and 11%, 12%, and 12% in modulus for samples T300/5208, C6000/PMR15 and C6000/P1700, respectively.

Results are shown for the gamma irradiated samples in Figs. 4-5. At the dose levels applied in these experiments, no large changes were observed in either stress or modulus. (The maximum difference from the control for any of the treatments was <10%).

To test for significant difference in the stress or modulus as a function of irradiation dosage, analysis of variance was done using a statistical analysis system (SAS) [10]. In a sample set the data for two irradiation treatments were compared at the 5% confidence levels using the Duncan Multiple range test. In all cases, the 5000 Mrad treatment was significantly different from the control for both the stress and the modulus. Earlier work has been reported by Parkinson and Sisman [11] on the effects of radiation on the mechanical properties of a number of plastics. Their work suggests that polymers containing aromatic rings are highly resistant to radiation and they attribute this to the absorption and dissipation of energy, without bond disruption, of aromatic rings. Using neutron and gamma radiation, they showed very little change in the mechanical properties of cured diamino-diphenyl methane epoxy and polyimide polymer at radiation dosages $> 10^9$ rads. Gamma irradiation experiments by Brown and O'Donnell [12] on aromatic polysulfone show that no deleterious effects occur in vacuum to dose levels of 600 Mrads. However, degradation of flexural properties did occur when irradiation of samples was done in air.

Bullock reported that fast-neutron irradiation of graphite fibers in air showed an increase in strength followed by a decrease (by as much as 25%) of

the control [13]. However, irradiation in a inert environment showed only an increase in the strength [14]. Jones and Peggs [15] show a small increase in both the strength and modulus of graphite when irradiated with fast neutrons at elevated temperature. In addition, they reported an increase in the crystallite dimension suggesting that the elevated temperature induced recrystallization.

Graphite fiber/epoxy composites irradiated in air at 75°C with a combination of gamma, fast neutrons and thermal neutrons showed a decrease in flexural strength and modulus [16]. When samples were irradiated in liquid N₂, increases in the strength and modulus were observed when tested at liquid N₂ temperature while a decrease in those parameters occurred when irradiated at liquid N₂ temperature but tested at room temperature.

The results reported here are consistent with earlier work on plastics, fibers and composites. All the composites that we have studied contain matrices which have an abundance of ring structures and none of the systems we studied showed any degradation in stress or modulus when subjected to ionizing radiation, under vacuum, to dose levels of 5×10^9 rads. The ring structures in the matrices and the fibers appear to protect the composite from radiation damage.

Conclusion:

Graphite fiber/epoxy, graphite fiber/polysulfone and graphite fiber/polyimide composites show no deleterious stress or modulus effects by the exposure of 0.5 Mev electron radiation in vacuum up to 5000 Mrad. At 5000 Mrad the stress and modulus increased by approximately 12% compared with the controls. Graphite fiber/epoxy composites show little change in stress and modulus when exposed to several hundred Mrad of gamma radiation. Therefore,

the results reported here indicate that graphite fiber composites would have a considerable lifetime in space (probably > 30 years) before strength and stiffness properties would be affected significantly by high energy radiation.

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The authors gratefully acknowledge NASA for support of the research, Dr. E. R. Long of NASA Langley Research Center for supplying samples and helpful suggestions, Dr. W. K. Walsh for help with irradiation experiments, Mr. W. C. Stuckey for help with the mechanical testing and to Dr. Vivian Stannett for his helpful comments.

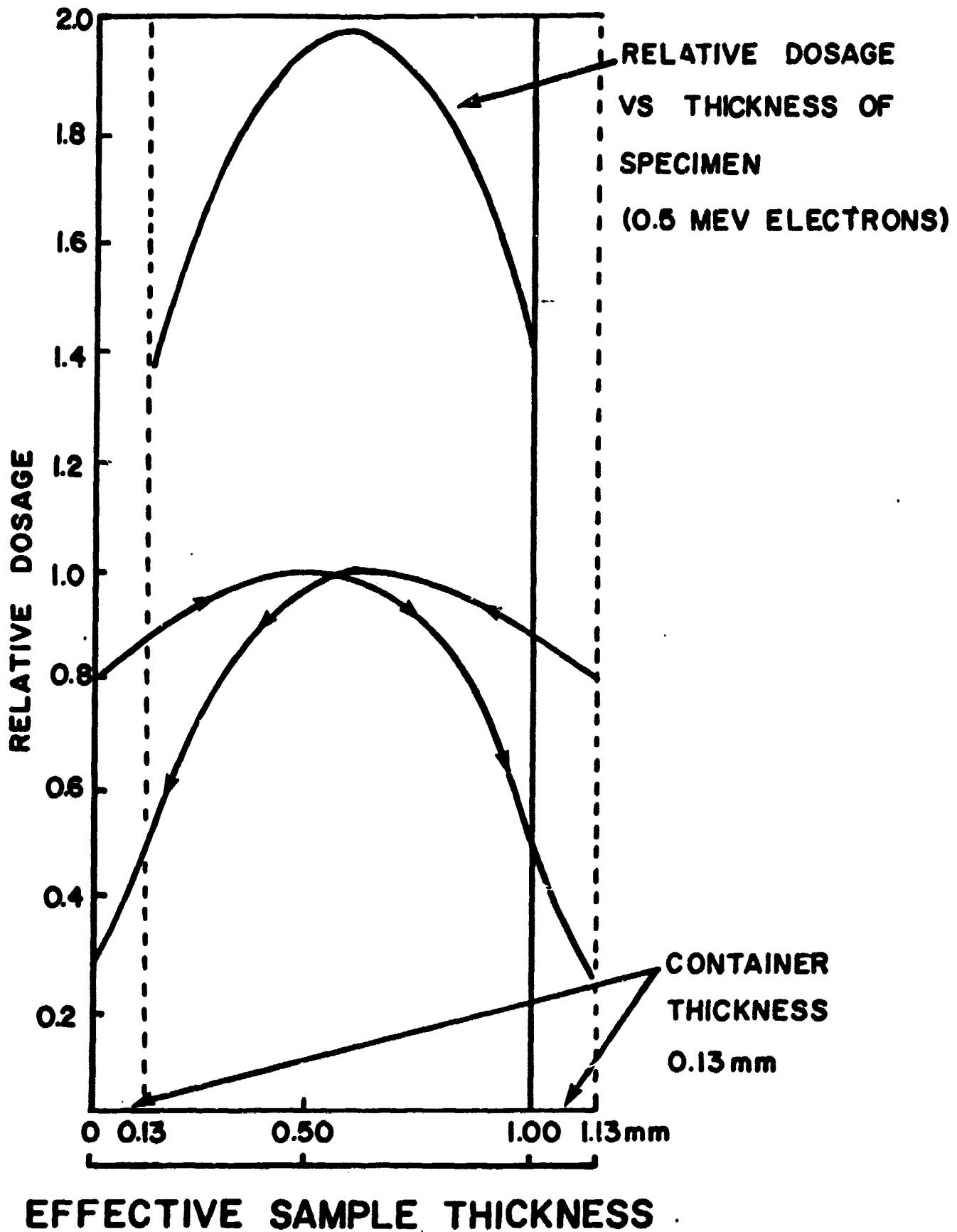
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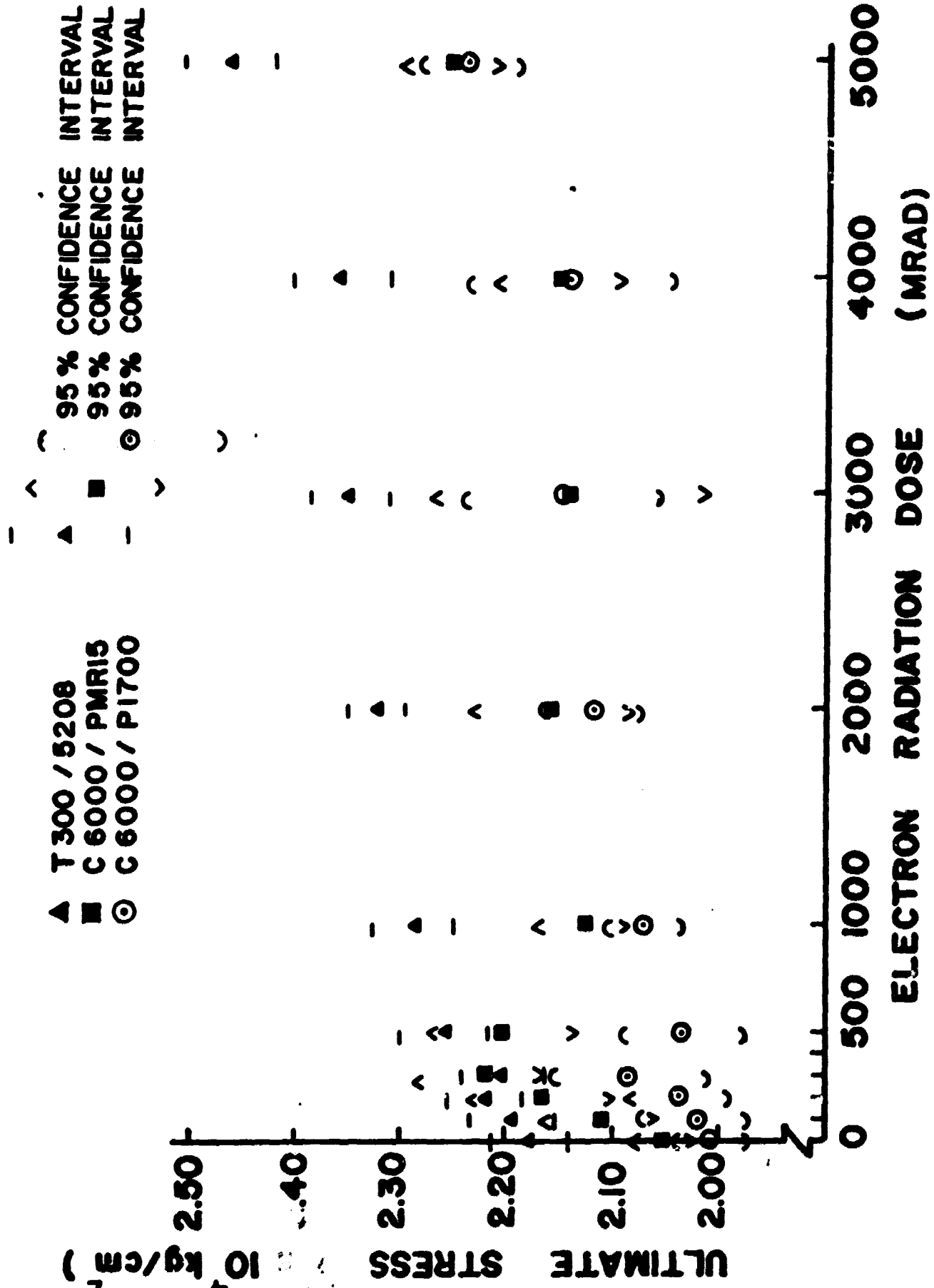
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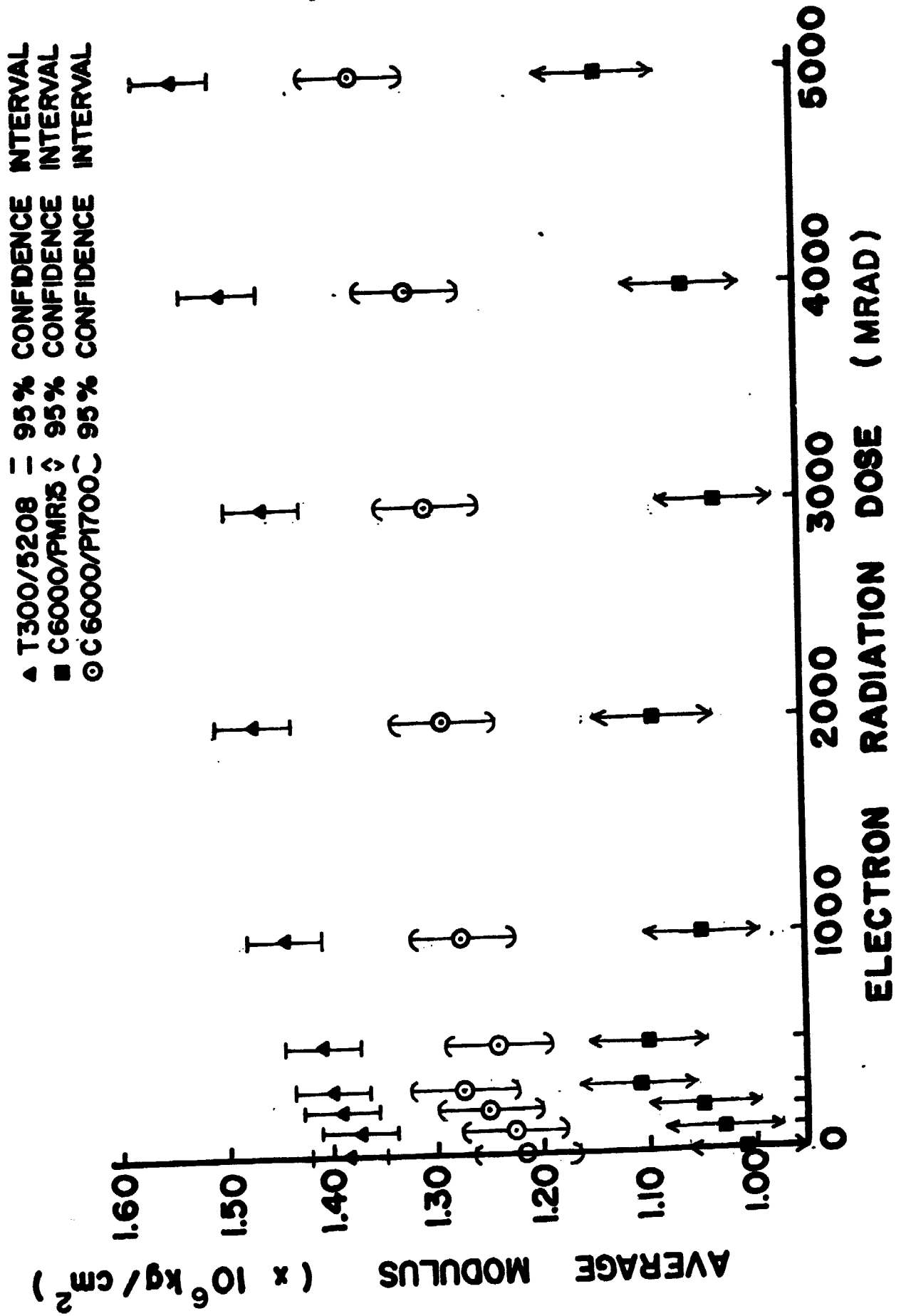
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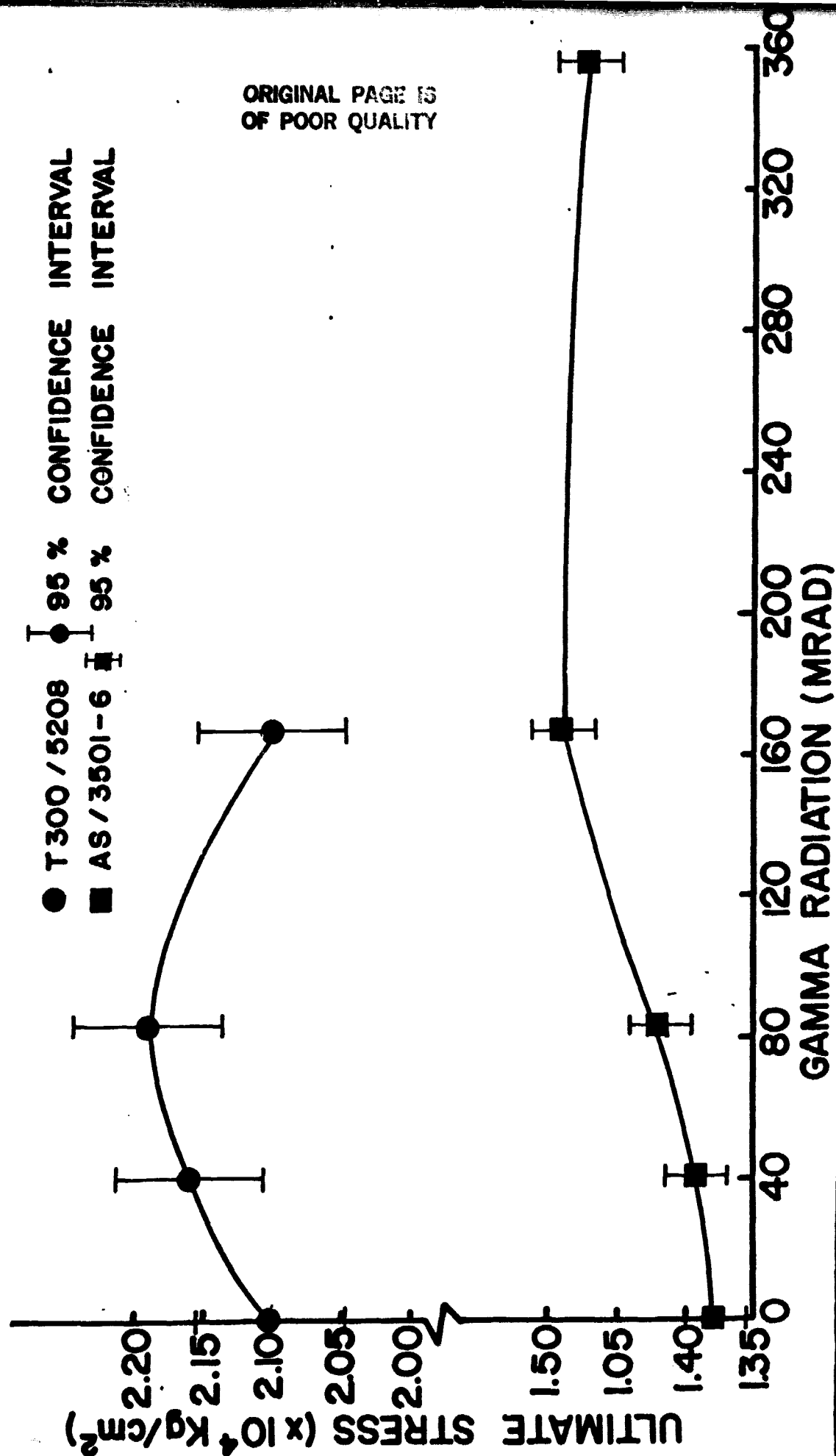
1. Estimate of relative composite sample dosage vs. thickness when exposed to 0.5 Mev electrons in the sample holders when both sides of sample are exposed to the beam. The dose-distance relationship is adjusted to unit density material by multiplying thickness by specific gravity.
2. Ultimate stress vs. 0.5 Mev electron irradiation dosage for graphite fiber composite samples.
3. Average modulus vs. 0.5 Mev electron irradiation dosage for graphite fiber composite samples.
4. Ultimate stress vs. 1.33 gamma irradiation dosage for graphite fiber/epoxy composites.
5. Average modulus vs. 1.33 gamma irradiation dosage for graphite fiber/epoxy composites.

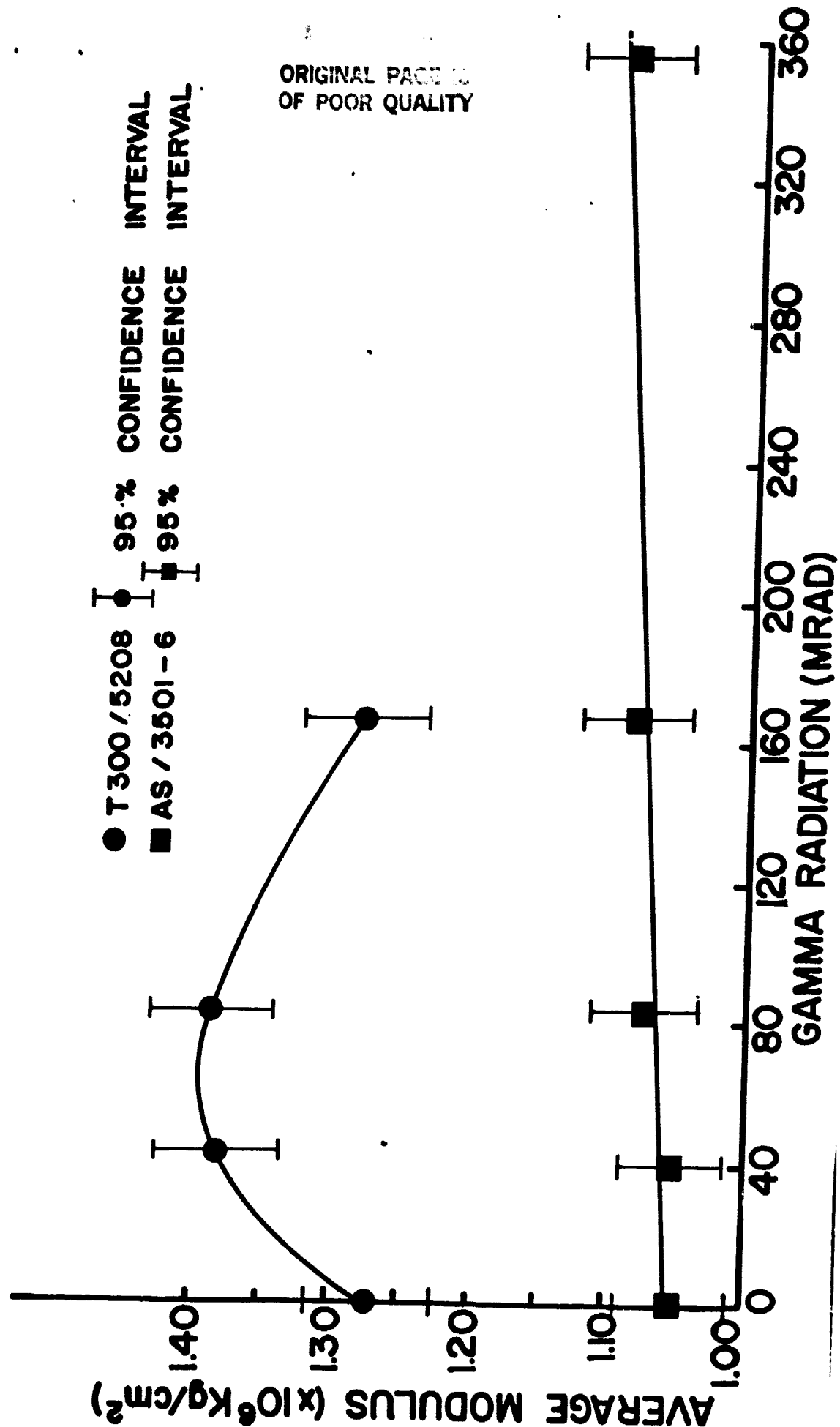






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Abstract Submitted
for the New York Meeting of the
American Physical Society

March, 1980

High Polymer Physics

Poster Session

Radiation Effects on the Mechanical Properties of Graphite Fiber Reinforced Composites. * R. E. FORNES, J. D. MEMORY, N. NARANONG, K. WOLF, and W. C. STUCKEY, N. C. State U. ** -- The effects of 1.33 Mev gamma and 0.5 Mev electron radiation on the ultimate stress and modulus of graphite fiber reinforced composite systems have been investigated. The fibers were uniaxial oriented in either an epoxy or polyimide matrix, fabricated into thin sheets, cured and irradiated under vacuum. The irradiation dosages ranged up to 800 Mrad for the electron irradiated samples and 300 Mrad for the gamma irradiated samples. The composites were tested using a three-point bending tester on an Instron. The tensile stress and modulus results indicated that no detrimental effects on the mechanical properties of the irradiated samples were observed. Both the stress and modulus tend to increase with maximum values of these parameters occurring around 200 Mrad.

*Submitted by R. E. Fornes.

**Supported by NASA Grant NSG-1562.

Prefer Poster Session

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Abstract Submitted
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American Physical Society

March, 1981

Condensed Matter
Number 60

Polymer Physics

Electron Spin Resonance Studies of Epoxy Samples
Exposed to 1/2 Mev Electrons. * K. SCHAFFER, R. D.
GILBERT, J. D. MEMORY, and R. E. FORNES, N. C.
State U. --Samples of cured epoxy resin (MY 720/DDS
Ciba Geigy) have been irradiated at room temperature
with 1/2 Mev electrons to dose levels up to 5×10^7 rads.
These samples were stored at cryogenic temperatures
following exposure until measurements were made on an
x-band ESR spectrometer. Under the conditions of meas-
urements, radical concentrations leveled off around 10^{19}
radicals/cm³. Before irradiation, cured samples showed
the presence of radicals with concentrations ranging from
 10^{17} - 10^{18} radicals/cm³. The spectra of both cured and
irradiated resins are asymmetric. Details of the results
will be given. Estimates of radical decay rates will also
be presented as well as ESR measurements at 77°K.
*Supported by NASA Grant NSG 1562.

Prefer Poster Session

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Polymer Physics

Effects of Ionizing Radiation on the Mechanical and Morphological Properties of Graphite Fiber Reinforced Composites. * N. NARANONG, K. WOLF, J. D. MEMORY, R. D. GILBERT, and R. E. FORNES, N. C. State U. --Several types of uniaxial composite samples have been irradiated with 1/2 Mev electrons to dose levels up to 5×10^9 rads. Strength and modulus results, based on three point bending tests, show that these materials withstand these levels of radiation with no deleterious effects. There is a small, approximately monotonic, increase in both ultimate stress and modulus over this range of radiation. In addition, ruptured samples have been investigated using scanning electron microscopy (SEM). These results show very little morphological differences between control and irradiated samples. Details of the mechanical tests and the SEM investigations will be reported.
*Supported by NASA Grant NSG 1562.

Prefer Standard Session

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